

question, $\sim 300 \text{ m}^2/\text{g}$, is made available by the mesopores. Because the total porosity of the monolithic silica matrix is higher than 80%, the user is able to perform his chromatography using a much lower back pressure than with conventional particles that have a total porosity of only app. 65%. By optimising the ratio of throughpores to total porosity and the silica gel skeleton thickness, separations become possible at higher flow rates. This is due to accelerated adsorption and desorption of the substances to be separated by the silica gel surface.

Due to better mass transfer properties of a monolithic skeleton over distinct particles, high-speed separation is possible without noticeable impact on resolution. For example, five β -blockers were separated at high α values ($\alpha = k'_{\text{Substance2}}/k'_{\text{Substance1}}$) and with excellent peak symmetry. At a flow rate of 1 ml/min, the 5 substances were separated within 5 minutes. Under these conditions, the total system backpressure was 13 bar. At a flow rate of 9 ml/min., the 5 β -blockers can be baseline separated within

half a minute with a column backpressure of only 72 bar (Fig. 2). The low backpressure resulting from the enhanced permeability of monolithic vs. particulate columns offers a number of additional benefits that chemists can profit from.

Using monolithic columns is a way to increase the column bed length—and therefore the absolute plate number. This is only possible due to the minimal backpressure generated by monolithic HPLC columns. Due to better mass transfer properties, high separation efficiency is maintained even at high linear flow rates. As a result, the sample throughput of an average laboratory can be increased easily.

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Electrophoretic Deposition (EPD) Coatings of Sol-Gel Solutions and Suspensions

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The use of metals is restricted in applications where aggressive conditions can lead to corrosion or wear. Different kinds of coatings have been studied to protect the metal surface, including metallic, polymeric, inorganic and, more recently, hybrid organic-inorganic coatings. Sol-gel has demonstrated to be a powerful route for obtaining glass-like coatings onto metals with suitable protecting properties. However, a major restriction of the sol-gel technology is the low thickness attainable with the mechanical depo-

sition techniques, like dipping, spin-coating and spraying.

One possibility for increasing the coating thickness is the addition of colloidal particles, typically 10–50 nm in size [1] or glass powder to the precursor solution [2]. A further improvement is the increase of the particles concentration in the deposit promoting the migration of particles through the suspension towards the substrate by electrophoresis [3].

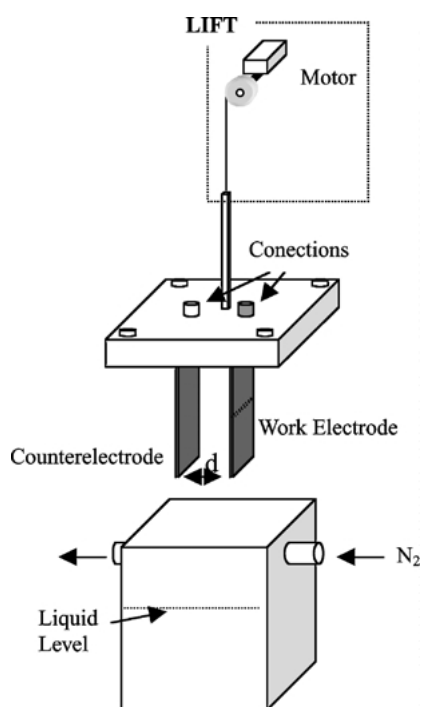


Fig. 1 Scheme of the equipment designed for EPD.

EPD is a powerful coating technique because it is extremely versatile, allowing a broad margin of thicknesses and complex shapes, being also low-cost and reliable process. Figure 1 shows a scheme of the equipment designed for EPD of sol-gel suspensions which allows to control the atmosphere and the withdrawal rate. This makes it possible to distinguish between the EPD and the dipping contributions to the coating thickness.

A basic requirement for EPD is that suspended particles are stable and have a high electrophoretic mobility [4].

The coating of stainless steel sheets by EPD was studied using two synthesis routes to obtain SiO_2 and $75\text{SiO}_2/25\text{ZrO}_2$ coatings. In a first colloidal synthesis route, a hybrid organic-inorganic sol was prepared from TEOS and MTEOS with acid catalysis and SiO_2 and ZrO_2 aqueous suspensions were further added. In a second

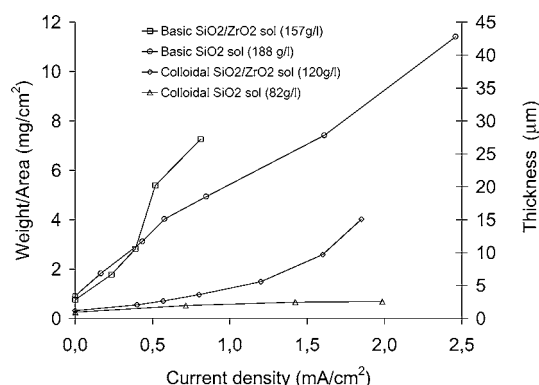


Fig. 2 Weight per unit area and thickness evolution of the deposits obtained by EPD as a function of current densities from different sols.

route, the sol was prepared by basic synthesis from mixing silica [5] or silica/zirconia precursors with NaOH and reacted by adding water.

Figure 2 plots the weight per unit area and the coating thickness for current densities ranging from 0.2 to 2.4 mA/cm^2 . Thickness increases with current density in all cases, but thicker coatings are produced from sols obtained by basic synthesis. Figure 3 shows the cross section of a 12 μm crack-free coating strongly adhered to the

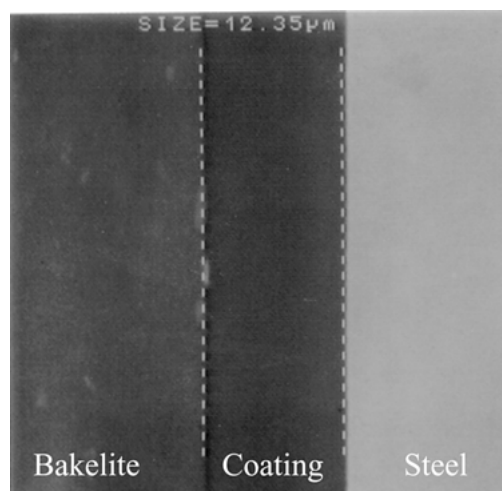


Fig. 3 Cross section of a 12 μm sintered coating obtained by EPD from a basic catalysed silica sol.

stainless steel, deposited at a current density of 0.6 mA/cm^2 during 30 minutes from a 188 gr/l SiO_2 basic sol and sintered at 500°C for 0.5 hours. The protective character of the film was verified by potentiodynamic tests performed on 1 cm^2 areas. The corrosion current density is reduced by four orders of magnitude, from 10^{-6} A/cm^2 for the uncoated steel to 10^{-10} A/cm^2 for the coated one. The pitting potential surpasses 1 V , demonstrating that the thick glass coating produced by EPD is an efficient barrier against electrolytic corrosion. This confirms that if any porosity still remains the pores are closed and non-connected.

In summary, EPD of particulate sol gel solutions and suspensions allows to obtain thicker coatings than conventional techniques, with high homogeneity and excellent corrosion resistance while maintaining similar qualities.

Acknowledgments

This work has been supported by CICYT (MAT99-0872) and EC BRITE Programme (BE97-5111) in collaboration with INM (Germany), Miele (Germany), Corus (The Netherlands), Ferro (France) and ABB (France). Dr. Mennig and Dr. Niegisch (INM) and Dr. A Conde and Dr. J.J. Damborenea (CENIM) are gratefully acknowledged for sol processing and potentiodynamic studies.

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